

CLAIMS:

1. A method for the production of a metal or metal alloy of interest which comprises electrochemically reducing an anode formed of a composite of a metal oxide of the metal of interest with carbon in a molten salt electrolyte.
- 5 2. The method of claim 1, wherein the anode is formed of a titanium oxide or titanium suboxide-carbon composite, and the metal produced comprises titanium.
3. The method of claim 1, wherein the anode is formed of a chromium oxide-carbon composite, and the metal produced comprises chromium.
4. The method of claim 1, wherein the anode is formed of a hafnium oxide-
10 carbon composite, and the metal produced comprises hafnium.
5. The method of claim 1, wherein the anode is formed of a molybdenum oxide-carbon composite, and the metal produced comprises molybdenum.
6. The method of claim 1, wherein the anode is formed of a niobium oxide-carbon composite, and the metal produced comprises niobium.
- 15 7. The method of claim 1, wherein the anode is formed of a tantalum oxide-carbon composite, and the metal produced comprises tantalum.
8. The method of claim 1, wherein the anode is formed of a tungsten oxide-carbon composite, and the metal produced comprises tungsten.
9. The method of claim 1, wherein the anode is formed of a vanadium oxide-
20 carbon composite, and the metal produced comprises vanadium.
10. The method of claim 1, wherein the anode is formed of zirconium oxide-carbon composite, and the metal produced comprises zirconium.

11. The method of claim 1, wherein said molten salt electrolyte comprises a strong Lewis acid.

12. The method of claim 11, wherein the electrolyte is selected from the group consisting of an eutectic of sodium chloride, lithium chloride and potassium chloride, an eutectic of potassium fluoride, sodium fluoride and lithium fluoride, an eutectic of sodium chloride, calcium chloride and potassium chloride, an eutectic of sodium chloride, magnesium chloride and sodium fluoride, and an eutectic of sodium chloride, potassium chloride and sodium fluoride.

13. The method of claim 1, wherein an electric current is applied in a pulsed manner.

14. The method of claim 1, wherein an electric current is in a pulsed, periodically reversed polarity.

15. The method of claim 1, wherein the anode comprises oxides of two or more metals, and the metal produced comprises an alloy of said two or more metals.

16. An electrolytic cell for production of a metal or metal alloy of interest, said cell comprising in combination:

a molten salt electrolyte disposed in a cell, said electrolyte comprising a strong Lewis acid; and

a cathode and an anode in contact with said electrolyte, wherein said anode is formed of a composite of an oxide of the metal of interest and carbon.

17. The cell of claim 16, wherein the anode is formed of a titanium oxide or titanium suboxide-carbon composite, and the metal produced comprises titanium.

18. The cell of claim 16, wherein the anode is formed of a chromium oxide-carbon composite, and the metal produced comprises chromium.

19. The cell of claim 16, wherein the anode is formed of a hafnium oxide-carbon composite, and the metal produced comprises hafnium.

5 20. The cell of claim 16, wherein the anode is formed of a molybdenum oxide-carbon composite, and the metal produced comprises molybdenum.

21. The cell of claim 16, wherein the anode is formed of a niobium oxide-carbon composite, and the metal produced comprises niobium.

22. The cell of claim 16, wherein the anode is formed of a tantalum oxide-carbon
10 composite, and the metal produced comprises tantalum.

23. The cell of claim 16, wherein the anode is formed of a tungsten oxide-carbon composite, and the metal produced comprises tungsten.

24. The cell of claim 16, wherein the anode is formed of a vanadium oxide-carbon composite, and the metal produced comprises vanadium.

15 25. The cell of claim 16, wherein the anode is formed of zirconium oxide-carbon composite, and the metal produced comprises zirconium.

26. The cell of claim 16, wherein the electrolyte is selected from the group consisting of an eutectic of sodium chloride, lithium chloride and potassium chloride, an eutectic of potassium fluoride, sodium fluoride and lithium fluoride, an eutectic of sodium
20 chloride, calcium chloride and potassium chloride, an eutectic of sodium chloride, magnesium chloride and sodium fluoride, and an eutectic of sodium chloride, potassium chloride and sodium fluoride.

27. The cell of claim 16, wherein the anode comprises oxides of two or more metals, and the metal produced comprises an alloy of said two or more of said metals.

28. The cell of claim 16, and further comprising a source of electric current connected to said cell.

5 29. The cell of claim 28, wherein said source of electric current is connected to said cell via a current controller.

30. The cell of claim 29, wherein the current controller is adapted to apply current in a pulsed manner.

31. The cell of claim 29, wherein the current controller is adapted to apply current
10 in a pulsed, periodically reversed polarity.

32. The cell of claim 16, wherein the anode comprises loose pieces of said metal oxide carbon composite contained within a porous basket disposed in said electrolyte.

33. The cell of claim 16, and further comprising a valved outlet adjacent a lower wall thereof.

15 34. The cell of claim 16, and further comprising a separator or diaphragm disposed between said anode and cathode.

35. The cell of claim 34, wherein the separator or diaphragm comprises porous alumina.

36. An anode for use in a molten salt electrolytic cell for electrolytic production of
20 a metal or metal alloy of interest comprising a composite of an oxide of the metal of interest with carbon, wherein the metal of interest comprises a multi-valence or high valence metal or metal alloy.

37. The anode of claim 36, comprising a titanium oxide or titanium suboxide-carbon composite.

38. The anode of claim 36, comprising a chromium oxide-carbon composite.

39. The anode of claim 36, comprising a hafnium oxide-carbon composite.

5 40. The anode of claim 36, comprising a molybdenum oxide-carbon composite.

41. The anode of claim 36, comprising a niobium oxide-carbon composite.

42. The anode of claim 36, comprising a tantalum oxide-carbon composite.

43. The anode of claim 36, comprising a tungsten oxide-carbon composite.

44. The anode of claim 36, comprising a vanadium oxide-carbon composite.

10 45. The anode of claim 36, comprising zirconium oxide-carbon composite.

46. The anode of claim 36, comprising oxides of two or more multi-valence or high valence metals, and carbon.

47. A molten salt electrolyte comprising a eutectic of sodium chloride, lithium chloride and potassium chloride.

15 48. A molten salt electrolyte comprising an eutectic of potassium fluoride, sodium fluoride and lithium fluoride.

49. A molten salt electrolyte comprising an eutectic of sodium chloride, calcium chloride and potassium chloride.

50. A metal or metal alloy produced by the process of claim 1 in particulate, flake
20 or solid form.

51. A metal or metal alloy as claimed in claim 50, wherein the metal produced is selected from the group consisting of titanium, chromium, hafnium, molybdenum, niobium, tantalum, tungsten, vanadium, zirconium and an alloy of one or more of said metals.

52. An anode for use in a molten salt electrolytic cell for electrolytic production of titanium comprising a titanium suboxide - carbon composite.

53. The anode as claimed in claim 52 wherein the titanium suboxide comprises TiO or Ti₂O₃.

5 54. A method for the production of purified titanium from rutile ore which comprises electrowinning from an anode formed of a mixture of titanium suboxide/carbon composite in a molten salt electrolyte.

55. The method of claim 54, wherein the molten salt electrolyte is selected from the group consisting of an eutectic of sodium chloride, lithium chloride and potassium
10 chloride, an eutectic of potassium fluoride, sodium fluoride and lithium fluoride, an eutectic of sodium chloride, calcium chloride and potassium chloride, an eutectic of sodium chloride, magnesium chloride and sodium fluoride, and an eutectic of sodium chloride, potassium chloride and sodium fluoride.

56. The method of claim 54, wherein an electric current is applied in a pulsed
15 manner.

57. The method of claim 54, wherein an electric current is in a pulsed, periodically reversed polarity.

58. The method of claim 54, wherein titanium suboxide is mixed with carbon in a ratio of at least 1:1.5 over stoichiometry.

20 59. The method of claim 54, wherein the titanium suboxide is mixed with carbon in a ratio of at least 1.1 over stoichiometry.

60. A method for the production of purified titanium which comprises electrochemically reducing an anode formed of a titanium suboxide/carbon composite in a molten salt electrolyte.

61. The method of claim 60, wherein the molten salt electrolyte is selected from the group consisting of an eutectic of sodium chloride, lithium chloride and potassium chloride, an eutectic of potassium fluoride, sodium fluoride and lithium fluoride, an eutectic of sodium chloride, calcium chloride and potassium chloride, an eutectic of sodium chloride, magnesium chloride and sodium fluoride, and an eutectic of sodium chloride, potassium chloride and sodium fluoride.

62. The method of claim 60, wherein an electric current is applied in a pulsed manner.

63. The method of claim 60, wherein an electric current is in a pulsed, periodically reversed polarity.

64. The method of claim 60, wherein titanium suboxide is mixed with carbon in a ratio of at least 1:1.5 over stoichiometry based on titanium.

65. The method of claim 60, wherein the titanium suboxide is mixed with carbon in a ratio of at least 1.1 over stoichiometry based on titanium.

66. A method for the direct production of a metal or metal alloy of interest in a particulate state which comprises subjecting an anode, formed of a composite of a metal oxide of the metal of interest with carbon, to electrolytic reduction in a cell containing a molten salt electrolyte.

67. The method of claim 66, wherein the anode is formed of a titanium oxide or titanium suboxide-carbon composite, and the metal produced comprises titanium.

68. The method of claim 66, wherein the anode is formed of a chromium oxide-carbon composite, and the metal produced comprises chromium.

69. The method of claim 66, wherein the anode is formed of a hafnium oxide-carbon composite, and the metal produced comprises hafnium.

5 70. The method of claim 66, wherein the anode is formed of a molybdenum oxide-carbon composite, and the metal produced comprises molybdenum.

71. The method of claim 66, wherein the anode is formed of a niobium oxide-carbon composite, and the metal produced comprises niobium.

72. The method of claim 66, wherein the anode is formed of a tantalum oxide-
10 carbon composite, and the metal produced comprises tantalum.

73. The method of claim 66, wherein the anode is formed of a tungsten oxide-carbon composite, and the metal produced comprises tungsten.

74. The method of claim 66, wherein the anode is formed of a vanadium oxide-carbon composite, and the metal produced comprises vanadium.

15 75. The method of claim 66, wherein the anode is formed of zirconium oxide-carbon composite, and the metal produced comprises zirconium.

76. The method of claim 66, wherein said molten salt electrolyte comprises a strong Lewis acid.

77. The method of claim 76, wherein the electrolyte is selected from the group
20 consisting of an eutectic of sodium chloride, lithium chloride and potassium chloride, an eutectic of potassium fluoride, sodium fluoride and lithium fluoride, an eutectic of sodium chloride, calcium chloride and potassium chloride, an eutectic of sodium chloride, magnesium

chloride and sodium fluoride, and an eutectic of sodium chloride, potassium chloride and sodium fluoride.

78. The method of claim 66, wherein an electric current is applied in a pulsed manner.

5 79. The method of claim 66, wherein an electric current is in a pulsed, periodically reversed polarity.

80. The method of claim 66, wherein the anode comprises oxides of two or more metals, and the metal produced comprises an alloy of said two or more metals.

10 81. A process for purification of rutile which comprises reacting rutile with carbon at an elevated temperature under an inert atmosphere.

82. The method of claim 81, wherein the temperature is in excess of 1200°C.

83. The method of claim 82, wherein the temperature is between 1200°C and 1850°C.

15 84. The method of claim 81, further comprising the steps of forming said purified rutile into an electrode and employing the resulting electrode in electrolytic process to produce purified titanium.

85. The method of claim 60, wherein titanium suboxide-carbon composite anode is formed by heating a titanium oxide with carbon under an inert atmosphere.

20 86. A method of production of purified titanium which comprises electrowinning titanium oxide in a molten salt of calcium fluoride at elevated temperature.

87. The method of claim 86, wherein the elevated temperature is above 1670°C.

88. The method of claim 87, wherein the temperature is above 1700°C.

89. The method according to claim 66, wherein the electrode is formed of a titanium oxide/carbon composite, and including the step of adding a Ti^{+2} containing compound to the electrolyte.

90. The method of claim 89, wherein the Ti^{+2} containing compound is added in a concentration of $\frac{1}{2}$ to 20 % by weight of the electrolyte.

91. The method of claim 90, wherein the Ti^{+2} containing compound is added in a concentration of 1 to 10 % by weight of the electrolyte.

92. The method according to claim 60, wherein the anode comprises a composite of titanium suboxide and carbon, and including the step of adding a Ti^{+2} containing compound to the electrolyte.

93. The method of claim 66, wherein the electrolyte includes a Ti^{+3} containing compound which is reduced in one step to titanium metal.

94. The method of claim 93, wherein the Ti^{+2} containing compound is added in a concentration of $\frac{1}{2}$ to 20 % by weight of the electrolyte.

95. The method of claim 94, wherein the Ti^{+2} containing compound is added in a concentration of 1 to 10 % by weight of the electrolyte.

96. A method for the production of a metal hydride of interest which comprises electrochemically reducing an anode, formed of a composite of a metal oxide of the metal of interest with carbon, in a molten salt electrolyte in the presence of hydrogen gas.

97. The method of claim 96, wherein the anode is formed of a titanium oxide or titanium suboxide-carbon composite, and the metal produced comprises titanium hydride.

98. The method of claim 96, wherein the anode is formed of a chromium oxide-carbon composite, and the metal produced comprises chromium hydride.

99. The method of claim 96, wherein the anode is formed of a hafnium oxide-carbon composite, and the metal produced comprises hafnium hydride.

100. The method of claim 96, wherein the anode is formed of a molybdenum oxide-carbon composite, and the metal produced comprises molybdenum hydride.

5 101. The method of claim 96, wherein the anode is formed of a niobium oxide-carbon composite, and the metal produced comprises niobium hydride.

102. The method of claim 96, wherein the anode is formed of a tantalum oxide-carbon composite, and the metal produced comprises tantalum hydride.

10 103. The method of claim 96, wherein the anode is formed of a tungsten oxide-carbon composite, and the metal produced comprises tungsten hydride.

104. The method of claim 96, wherein the anode is formed of a vanadium oxide-carbon composite, and the metal produced comprises vanadium hydride.

105. The method of claim 96, wherein the anode is formed of zirconium oxide-carbon composite, and the metal produced comprises zirconium hydride.

15 106. A molten salt electrolyte comprising a mixture of fluorine salt and a chlorine salt in a fluorine/chlorine ratio of at least 0.1 for use in producing titanium by electrowinning.

107. A method for the production of titanium metal which comprises electrochemically reducing a cathode formed of a titanium suboxide-carbon composite in a fused salt electrolyte.

20 108. The method of claim 107, wherein the fused salt electrolyte comprises calcium chloride.

109. The method of claim 108, wherein the fused salt electrolyte contains calcium oxide.